

BEST AVAILABLE COPY



**PCT**

WORLD INTELLECTUAL PROPERTY ORGANIZATION  
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification 6 : <b>D21H 21/24, 17/10, D06M 13/292, 13/44, 13/165, 15/53</b></p>	<p><b>A1</b></p>	<p>(11) International Publication Number: <b>WO 97/31153</b></p> <p>(43) International Publication Date: 28 August 1997 (28.08.97)</p>		
<table style="width: 100%; border: none;"> <tr> <td style="width: 50%; border: none; vertical-align: top;"> <p>(21) International Application Number: <b>PCT/US97/02423</b></p> <p>(22) International Filing Date: 18 February 1997 (18.02.97)</p> <p>(30) Priority Data: 03/604,818                      23 February 1996 (23.02.96)      US</p> <p>(71) Applicant: <b>QUAKER CHEMICAL CORPORATION</b> [US/US]: Elm &amp; Lee Streets, Conshohocken, PA 19428-0809 (US).</p> <p>(72) Inventors: <b>EVANS, Robert, D.</b>; 309 Parry Road, Warminster, PA 18974-4033 (US). <b>MORRISON, Suzanne</b>; 2 West Merion Circle, Downingtown, PA 19335 (US). <b>CAMPBELL, Clayton</b>; 443 Spruce Drive, Exton, PA 19341 (US).</p> <p>(74) Agents: <b>DONATIELLO, Guy, T. et al.</b>; Ratner &amp; Prestia, Suite 301, One Wesilakes, Berwyn, P.O. Box 980, Valley Forge, PA 19482-0980 (US).</p> </td> <td style="width: 50%; border: none; vertical-align: top;"> <p>(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).</p> <p><b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p> </td> </tr> </table>			<p>(21) International Application Number: <b>PCT/US97/02423</b></p> <p>(22) International Filing Date: 18 February 1997 (18.02.97)</p> <p>(30) Priority Data: 03/604,818                      23 February 1996 (23.02.96)      US</p> <p>(71) Applicant: <b>QUAKER CHEMICAL CORPORATION</b> [US/US]: Elm &amp; Lee Streets, Conshohocken, PA 19428-0809 (US).</p> <p>(72) Inventors: <b>EVANS, Robert, D.</b>; 309 Parry Road, Warminster, PA 18974-4033 (US). <b>MORRISON, Suzanne</b>; 2 West Merion Circle, Downingtown, PA 19335 (US). <b>CAMPBELL, Clayton</b>; 443 Spruce Drive, Exton, PA 19341 (US).</p> <p>(74) Agents: <b>DONATIELLO, Guy, T. et al.</b>; Ratner &amp; Prestia, Suite 301, One Wesilakes, Berwyn, P.O. Box 980, Valley Forge, PA 19482-0980 (US).</p>	<p>(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).</p> <p><b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>
<p>(21) International Application Number: <b>PCT/US97/02423</b></p> <p>(22) International Filing Date: 18 February 1997 (18.02.97)</p> <p>(30) Priority Data: 03/604,818                      23 February 1996 (23.02.96)      US</p> <p>(71) Applicant: <b>QUAKER CHEMICAL CORPORATION</b> [US/US]: Elm &amp; Lee Streets, Conshohocken, PA 19428-0809 (US).</p> <p>(72) Inventors: <b>EVANS, Robert, D.</b>; 309 Parry Road, Warminster, PA 18974-4033 (US). <b>MORRISON, Suzanne</b>; 2 West Merion Circle, Downingtown, PA 19335 (US). <b>CAMPBELL, Clayton</b>; 443 Spruce Drive, Exton, PA 19341 (US).</p> <p>(74) Agents: <b>DONATIELLO, Guy, T. et al.</b>; Ratner &amp; Prestia, Suite 301, One Wesilakes, Berwyn, P.O. Box 980, Valley Forge, PA 19482-0980 (US).</p>	<p>(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).</p> <p><b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>			
<p>(54) Title: <b>MANUFACTURE OF SOFTENED CELLULOSE FIBER-BASED PRODUCTS</b></p> <p>(57) Abstract</p> <p>A process and composition for softening cellulose fibers for the manufacture of softened cellulose fiber-based products such as paper towels, facial tissue, sanitary tissues, toilet tissues, non-woven fabrics and fluff pulp, and the products made by said process. The process maintains a high level of wet strength in the finished paper product containing suitable wet strength additives, as measured by the wet tensile strength of the dry web. The process includes treating cellulose fiber with chemical softening composition comprising phospholipids, a non-ionic surfactant and, optionally, a lubricating additive. The composition includes a phospholipid, a non-ionic surfactant and, optionally, a lubricant. The products include softened cellulose fiber-based material including a composition which includes a phospholipid, a non-ionic surfactant and, optionally, a lubricant. The phospholipids include phosphatidylcholine (lecithin), hydroxylated phosphatidylcholine, phosphatidylethanolamine, hydroxylated phosphatidylethanolamine, phosphatidylserine, hydroxylated phosphatidylserine, phosphatidylinositol and hydroxylated phosphatidylinositol.</p>				

**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AM	Armenia	GB	United Kingdom	MW	Malawi
AT	Austria	GE	Georgia	MX	Mexico
AU	Australia	GN	Guinea	NE	Niger
BB	Barbados	GR	Greece	NL	Netherlands
BE	Belgium	HU	Hungary	NO	Norway
BF	Burkina Faso	IE	Ireland	NZ	New Zealand
BG	Bulgaria	IT	Italy	PL	Poland
BJ	Benin	JP	Japan	PT	Portugal
BR	Brazil	KE	Kenya	RO	Romania
BY	Belarus	KG	Kyrgyzstan	RU	Russian Federation
CA	Canada	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic	KR	Republic of Korea	SE	Sweden
CG	Congo	KZ	Kazakhstan	SG	Singapore
CH	Switzerland	LI	Liechtenstein	SI	Slovenia
CI	Côte d'Ivoire	LK	Sri Lanka	SK	Slovakia
CM	Cameroon	LR	Liberia	SN	Senegal
CN	China	LT	Lithuania	SZ	Swaziland
CS	Czechoslovakia	LU	Luxembourg	TD	Chad
CZ	Czech Republic	LV	Latvia	TG	Togo
DE	Germany	MC	Monaco	TJ	Tajikistan
DK	Denmark	MD	Republic of Moldova	TT	Trinidad and Tobago
EE	Estonia	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	UG	Uganda
FI	Finland	MN	Mongolia	US	United States of America
FR	France	MR	Mauritania	UZ	Uzbekistan
GA	Gabon			VN	Viet Nam

**MANUFACTURE OF SOFTENED CELLULOSE FIBER-BASED PRODUCTS****Field of the Invention**

5           This invention relates to the manufacture of softened cellulose fiber-based products. More specifically, it relates to soft, absorbent cellulose fiber-based products based on nonwoven webs, air laid nonwoven fabrics, wet laid nonwoven fabrics, wet laid paper webs, and dry laid paper used to manufacture products such as toweling, sanitary (bath) tissue, facial tissue products, diaper cores, feminine care products, and  
10       cover stock for health care products.

**Background of the Invention**

          With certain softened cellulose fiber-based products manufactured today, and commonly used in such items as paper towels, facial tissue, sanitary tissues, toilet tissues, nonwoven fabrics and fluff pulp, three physical attributes are particularly  
15       important for their effective use. These attributes are strength, softness, and absorbency. Softness is the tactile sensation perceived by the consumer that is influenced by a combination of several physical properties. These include the stiffness, tensile strength of the paper, sheet bulk, and the surface feel or surface friction properties of the paper. Fluff pulp properties are measured by mullen energy and  
20       kamus energy.

          Absorbency is the measure of the ability of a product and of the fiber base from which the product may be made, to absorb quantities of liquid, particularly aqueous

liquids. Overall, absorbency as perceived by the consumer is generally considered to be a combination of the total quantity of liquid a given mass of cellulose-based material will absorb at saturation, and the rate at which the mass absorbs the liquid. With certain products such as paper towels, facial tissue, sanitary napkins and diapers, the absorbency of the product with aqueous liquid is most important.

Many methods and chemical agents have been developed for imparting softness to paper and paper products. Shaw, in US Patent #3,821,068, issued June 28, 1974, teaches that chemical debonders can be used to reduce the stiffness and thus enhance the softness of a tissue paper web. Becker et al., in US Patent #4,158,594, issued June 19, 1979, teaches a method for enhancing the bulk, softening and strength characteristics of tissue paper and paper towels softened by adding chemical debonding agents (which by their nature serve to weaken interfiber bonds within the web) to the paper making process. The '594 patent also teaches, optionally, creping the web (after debonding the web) by adhering one surface of the web to a creping surface in a fine pattern arrangement with a bonding material such as a water soluble adhesive. The bonding material is adhered to one surface of the web and to the creping surface in the fine pattern arrangement and then the web is creped from the creping surface to form a sheet material.

Chemical debonding agents have been disclosed in many other references such as US Patent #3,554,862, issued to Harvey et al. on January 12, 1971. These materials include quaternary ammonium salts such as trimethylcocoammonium chloride, trimethyloleylammonium chloride, dimethyldi (hydrogenated tallow) ammonium

chloride, and trimethylstearyl ammonium chloride. The '862 patent teaches that while the addition of debonding agents to tissue paper webs enhances the softness of the webs, the debonding agents have been shown to decrease the absorbency of the webs. Emanuelson et al., in US Patent #4,144,122, issued March 13, 1979, teaches  
5 the use of complex quaternary ammonium compounds, such as bis (alkoxy- (2-hydroxy) - propylene) quaternary ammonium chlorides to soften webs, and overcome the absorbency decrease problem with the use of non-ionic surfactants such as ethylene oxide and propylene oxide adducts of fatty alcohols.

Drach et al. (US Patent #4,720,383, issued 1/19/88) teaches the use of novel  
10 quaternary imidazolinium compounds in combination with non-ionic polyoxyethylene and polyoxypropylene surfactants, to obtain paper products having good softness and with very little strength reduction.

Armak Company (of Chicago, IL, in their bulletin 76-17 (1977)) teaches that the use of dimethyl di (hydrogenated tallow) ammonium chloride in combination with fatty  
15 acid esters of polyoxyethylene glycols to impart both softness and absorbency to tissue paper webs.

It is apparent from much of the prior teachings that many different types of quaternary ammonium compounds can be used alone or in combinations with various non-ionic surfactants, to impart softness properties to cellulose fiber-based products,  
20 while at the same time minimizing the detrimental effects on absorbency. The cationic nature of these quaternary ammonium compounds makes them well suited for addition to paper prior to formation of the paper web when the cellulose is dispersed in water.

Such compounds also can be effectively applied to the cellulose after the formation of a paper web.

It is known that paper products, such as facial and sanitary tissues, and especially paper toweling, are used for many different functions in the common household. For example, paper towels are often used for wrapping and storing food. They are also used extensively in microwave cooking where the product is used to cover, and/or serves as an absorbent layer under, the food being cooked. In many cases, the paper product comes in direct contact with the food, and the paper manufacturers must consider the nature of the chemicals that are included in the paper product. For this reason, much attention and concern is currently given to the chemical agents used in the production of these paper products relative to their toxicity, irritating tendencies, as well as their tendencies to be washed or leached out of the paper product during use. There is a strong need in the industry, therefore, for softener technology which meets these demands, and in many cases, there is a strong desire in the industry for chemical softener agents which consist solely of materials which are considered to be completely safe for use in paper products for direct contact with food. Examples of compounds considered to be approved for use with foods are described and listed in the US FDA paragraphs 176.170, 176.180, and 176.210. While certainly known to be effective chemical softeners, none of the quaternary ammonium compounds previously described for use as softeners meet the current demands for approval under the appropriate FDA paragraphs. Therefore, while not a limiting factor of the present invention, there is a strong need in the industry for new chemical

softening technology capable of meeting the demands and concerns which currently exist.

Similar concerns exist for softened cellulose fiber-based products used for direct skin contact. Diapers and feminine hygiene products are examples of such products.

5 Recently, various new chemical softening methods have been described which do not involve quaternary ammonium compounds for their use. Spindel (US Patent #4,940,513, issued 7/10/90) teaches the use of a process for making soft tissue paper which includes the steps of wet-laying cellulose fibers to form a web, applying to the wet web a non-cationic surfactant, and drying and creping to form the finished tissue  
10 paper. It is described that because of the lack of ionic attraction for the cellulose fibers, the non-cationic materials need to be applied subsequent to formation of the wet web materials and prior to drying the web to completion. The non-cationic materials described include anionic surfactants such as alkali metal soaps of higher fatty acids, as well as alkali metal sulfates and sulfonates, non-ionic surfactants, which are  
15 described extensively in US Patent #4,940,513, columns 7-10, ampholytic surfactants, as well as zwitterionic surfactants of the type described in detail in US Patent #4,959,125, columns 11-12, as well as in US Patent #3,929,678, columns 18-22.

A different approach to chemical softeners is described in US Patent #5,059,282 and US Patent #5,164,046, which teach of the use of application of polysiloxanes to the  
20 paper web to improve the tactile softness of the final paper product.

Kinsley in WO 93/16229, WO 95/04856, and WO 95/04857 describes a method for increasing the strength of paper by first preparing a slurry of a cellulosic pulp, a

particulate binder substantially insoluble in water, and an emulsion comprised of lecithin and a fatty acid or derivative thereof. The applications describe the formation of strengthened paper products using a polyvinyl alcohol binder. The invention is not directed to softness or softened cellulose based products.

5           Nikoloff et al. in U.S. Patent 4,766,015 describe a lubricant additive mixture for use in combination with an aqueous-based paper or paperboard coating material where phospholipids such as lecithins are added to a coating material before the coating is applied to paper or paperboard. The paper is coated to improve properties such as appearance and printability. The web and the process described do not relate to a  
10           softened cellulose fiber-based material.

          While the newer chemical softener technology just described is free of quaternary ammonium compounds, there still exist important limitations particularly in that application of the non-cationic softeners must be done directly onto the formed paper web and cannot be accomplished by addition of the softening agent to a water  
15           dispersion of the cellulose before it is wet laid into a web. Such an application site is often desirable and can give beneficial properties relative to strength and softness of the final paper product. Also many of the non-cationic materials described in the prior art would not alleviate the concerns for safety when the material is in direct contact with food. Such materials are not listed on the appropriate paragraphs of the FDA.  
20           Therefore, there still exists a need for new softener technology which can be equally effective in producing soft paper tissue and toweling when applied either to a wet-laid web or to a water dispersion of the cellulose before formation of the paper web, which

is also effective in giving the desired sheet properties when applied with commonly used wet and dry strength additives, and which is comprised of materials all of which are considered safe for use in paper products for direct contact with food stuff as described by FDA paragraphs 176.170, 176.180 and 176.210. The current invention

5 meets these needs and offers new chemical softener technology compatible with the many requirements and uses of today's manufactured paper tissues and towels.

### Summary of the Invention

The present invention includes a process and a composition for softening cellulose fibers for the manufacture of softened cellulose fiber-based products such as paper towels, facial tissue, sanitary tissues, toilet tissues, non-woven fabrics and fluff pulp and the products thereof. This process includes treating the cellulose fibers with a composition including a phospholipid, a non-ionic surfactant and, optionally, a lubricating additive. The composition includes a phospholipid, a non-ionic surfactant, and, optionally, a lubricating additive. At least some of the composition is retained on the cellulose fibers after any subsequent processing operations such as drying or creping. The present invention also includes softened cellulose fiber-based products which include a composition comprising a phospholipid, a non-ionic surfactant, and optionally, a lubricating additive.

The phospholipids include phosphatidylcholine (lecithin), hydroxylated phosphatidylcholine, phosphatidylethanolamine, hydroxylated phosphatidylethanolamine, phosphatidylserine, hydroxylated phosphatidylserine, phosphatidylinositol and hydroxylated phosphatidylinositol. As used herein, the term lecithins meant to encompass both lecithin and hydroxylated lecithin. The non-ionic surfactants include polyethylene glycol dioleate, polyethylene glycol dilaurate, polypropylene glycol dioleate, polypropylene glycol dilaurate, polyethylene glycol monooleate, polyethylene glycol monolaurate, polypropylene glycol monooleate and polypropylene glycol monolaurate. The lubricants include castor oil, olive oil,

hexadecanol, methyl tallowate, glycerol, glycerol monostearate, lanolin, decanol and octadecanol.

### Detailed Description of the Invention

5           The present invention encompasses a process for softening cellulose fibers for the manufacture of softened cellulose fiber-based products such as paper towels, facial tissue, sanitary tissues, toilet tissues, non-woven fabrics and fluff pulp, and the products made by the process. The process also allows for maintaining a high level of wet strength in finished paper containing suitable wet strength additives, as measured  
10           by the wet tensile strength of the dry web. The invention includes the addition of a chemical softening composition comprising phospholipids, a non-ionic surfactant and, optionally, a lubricating additive, at levels suitable to give the desired properties to the softened cellulose fiber-based product.

          Stages of the production process suitable for addition of said chemical softening  
15           composition to the cellulose fibers include those where the cellulose fibers are in an aqueous dispersion, such as the head box of the paper machine, the machine chest or stuff box, sites of addition subsequent to the formation of a wet-laid web but prior to drying, and also those sites of addition during or subsequent to final drying of the web. The chemical softening compound may be applied to a non-woven web prior to  
20           bonding, after bonding, for example, and during debonding in a pin or hammer mill. The chemical softening compound may also be applied during debonding in a disk refiner. The chemical softening compound is added to the cellulose fibers in an amount

of up to about 40 pounds phospholipid per ton of dry pulp. The non-ionic surfactant is also added to the cellulose fibers in an amount of up to about 40 pounds surfactant per ton of dry pulp.

It has been found in the current invention that phospholipids, and compositions  
5 containing such compounds are retained on the fiber at high levels when applied to wet towel or tissue paper subsequent to wet web formation. It has also been found that the compositions of this invention have high retention when added to aqueous dispersions of cellulose fiber prior to further processing. In order to add the softening composition at such stages of cellulose fiber production, the softening compositions  
10 must have high enough degree of attraction for the fibers to provide adequate retention of the softener on the subsequently formed softened cellulose fiber-based product. It has been found that phospholipids show a relatively high degree of attraction for the cellulose fibers when added to either aqueous dispersions of the cellulose fibers, or to a wet-laid cellulose web. It has also been found, and serves as a principle basis for the  
15 utility of this invention, that the combination of phospholipids, with certain non-ionic surfactants as well as hydrophobic lubrication additives, show a high level of retention on cellulose fibers when added to either an aqueous dispersion of the fibers or to a wet-laid web.

The softening composition can also be effectively applied to the cellulose fibers  
20 during the actual drying process or subsequent to the drying process, for example, when final dry paper sheet has been formed. The addition of the composition during the drying process can be made by spraying the softening composition onto the

Yankee cylinder which serves to dry the wet-laid web. Such additions effectively can be made alone or in combination with additions of the commonly used chemicals applied to Yankee driers, such as release additives and adhesives.

In considering the effectiveness of chemical softening compositions for use in the manufacture of softened cellulose fiber-based products such as softened tissue paper and paper toweling, certain properties of the composition as well as the produced paper must be addressed. The first property is the retention of the softener composition to the cellulose fibers when added to either an aqueous dispersion of cellulose fibers, a wet-laid web, or a dried paper web. To obtain optimum efficiency with the softener composition, as well as to ensure that as much of the softener composition as possible goes onto the cellulose fibers, a degree of attraction of the softener to the cellulose fibers is required. This attraction can be one of an ionic nature or one governed by the surface energy and colloidal properties of the softener. It is well known that cationic softening agents, which are described numerous in the prior art, have an ionic attraction to cellulose, and are capable of being highly retained on the fibers when added to either aqueous dispersions of cellulose fibers or to a wet-laid web. Non-cationic softening agents have also been described as having the ability to be retained on cellulose fibers when applied directly to a wet-laid web subsequent to its formation and prior to drying. The chemical softening composition described in the current invention has been found to have a high degree of retention on cellulose fibers when added to aqueous dispersions of cellulose, wet-laid webs prior to drying, as well

as to the wet-laid webs during or subsequent to drying. This is a very beneficial property and an unexpected discovery.

Phospholipids, which are structurally very well suited for imparting softness properties to softened cellulose fiber-based products, have been found according to the current invention, to have a high attraction for cellulose fibers and can be used as the primary component of a chemical softening composition. Also, a very desirable aspect of the current invention is the high retention obtained when using combinations of phospholipids with certain non-ionic surfactants, and optionally with certain lubricating additives. The combination of these materials in a chemical softening composition allows for the effective use of such chemical softening compositions whereby much of the desired softening and absorbency properties imparted to the final paper product are obtained from the non-ionic surfactants and lubricating additives, and the high degree retention of such materials is achieved by their addition in combination with phospholipids. Such behavior enables the addition of the chemical softening composition to the cellulose fibers at desirable sites of addition, such as those where the cellulose fibers are in an aqueous dispersion. Such sites of addition are not suitable when applying such non-ionic surfactants and lubricating additives alone.

It is believed that the effective retention of the non-ionic surfactant and lubricating additive components of the softening composition described in this invention, when such a composition is added to an aqueous dispersion of cellulose fibers, is due to the formation of mixed component micelles. The micelles which exist in the aqueous system are comprised of mixtures of the ionic phospholipids with non-ionic

surfactants and lubricating additives, and whereby the ionic nature of the phospholipid gives high attraction to the cellulose fibers. The aggregation of the non-ionic surfactants, and lubricating additives with the phospholipid, leads to a high "carry on" or retention of the non-ionic components to the cellulose fibers. Such behavior, while  
5 having desirable benefits regarding retention of the non-ionic components, also has been found to lead to an enhanced, synergistic behavior from the combination of the phospholipids with the selected non-ionic surfactants. Such enhanced performance from the composition relative to the individual components was unexpected, and is a desirable result obtained within the development of the current invention.

10 Another important requirement of a chemical softening agent used for the manufacture of tissue paper and paper towels is the ability to reduce the rigidity or bulk strength properties of the dry sheet, but also at the same time, only minimally reducing the wet tensile strength properties of the paper sheet. The ability to retain a degree of strength when wet is critical for effective performance of tissue paper and toweling, and  
15 while certain bulk properties of the paper must be modified to achieve a degree of softness, any effects of the chemical softening composition which produce a large reduction in the wet tensile strength of the paper tissue or towels produced are undesirable.

The method of the current invention provides a chemical softening composition  
20 that has been found to effectively increase the flexibility and softness of the sheet while producing reduced detrimental effects on the sheet's wet tensile strength relative to the chemical softening compositions currently known in the art. Such properties can be

achieved using the chemical softening composition described in this invention, when applied to cellulose which also contains various common wet and dry strength additives. Such additives, which are typically polymeric materials, are commonly added to cellulose fibers to impart a degree of wet or dry strength to the final paper product.

5 Such polymers include carboxymethyl cellulose polymers of various molecular weights, as well as commonly used cross-linked polyamide resins.

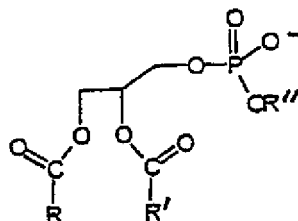
Conventionally pressed tissue paper, paper towels, and methods for making such paper are well known in the art. Such paper is typically made by depositing papermaking furnish on a foraminous forming wire. An example of one such wire  
10 commonly used in the art is a Fourdrinier wire. Once the furnish is deposited on the forming wire, it is referred to as a web. The web is dewatered by pressing the web and drying it at elevated temperature. The specific techniques and equipment for making webs according to the process just described are well known to those of ordinary skill in the art. In a typical process, a low consistency pulp furnish is provided in a pressurized  
15 head box. The head box has an opening for delivering a thin deposit of pulp furnish onto the Fourdrinier wire to form a wet web. The web is then typically dewatered to a fiber consistency of between about 7% and 25% water by weight by means of vacuum dewatering and further drying by pressing operations wherein the web is subjected to pressure developed by opposing cylindrical rolls. The dewatered web is then further  
20 pressed and dried by a steam apparatus known in the art as a Yankee dryer. Pressure can be developed at the Yankee dryer by a mechanical means. Multiple Yankee dryer drums may be used. The tissue or toweling paper structures which are formed are

referred to as conventional pressed structures. Such sheets are considered to be compacted since the web is subjected to substantial mechanical compressional forces while the fibers are moist and then dried while in a compressed state. Dewatering is commonly achieved for nonwoven materials and fluff pulp on a can drier or by air  
5 drying. In such cases dewatering is achieved to 7% to 40% water by weight.

The softened cellulose fiber-based products for which the present invention is useful includes but is not limited to fibers derived from wood, cotton, bagasse, hemp, straw and kenaf. Wood pulps for which the current invention is useful include but are not limited to kraft, mechanical and sulfite pulps. The source of the wood pulp can be  
10 from deciduous trees, often referred to as hardwoods, or from coniferous trees, which are typically referred to as softwoods, or from any combination of the two types. The present invention is useful for application to virgin pulp, recycled pulp, and mixtures thereof.

The phospholipids useful for the current invention include but are not limited to  
15 those derived from soya or other naturally occurring oils. The present invention includes in the softener composition from about 10 to about 85 percent by weight of a phospholipid or a mixture of phospholipids. The hydroxylated phospholipids are those which have been modified by hydroxylation. Such materials are typically water dispersable fluids with an HLB value of between 6-18. The HLB value refers to the  
20 hydrophobic to lipophilic balance of a material, and is generally a good indicator of the water dispersability of a given material. A specific phospholipid most preferred for use in the present invention is Centrolene A, a hydroxylated lecithin supplied by Central

Soya Company, Inc., of Fort Wayne, Indiana. This material is a water dispersable hydroxylated lecithin with an HLB value of 10, and a maximum acid value of 30. While marketed as a lecithin it does, in fact, contain a mixture of phospholipids that are well known to be present in soya. While this hydroxylated lecithin is preferred for use in the current invention, other phospholipids are also suitable for use. The general structure of the phospholipids useful within the scope of the current invention is shown in Formula 1,



Formula 1

- wherein R and R' are independently selected from the group consisting of
- (i) a substituted or unsubstituted, saturated or unsaturated, linear, branched or cyclic hydrocarbon chain of 6 to 22 carbons; and
  - (ii) a substituted or unsubstituted, saturated or unsaturated, linear, branched or cyclic hydrocarbon chain of 6 to 22 carbons including at least one heteroatom selected from the group consisting of nitrogen, oxygen, sulfur, phosphorus and halogen; and R'' contains at least one positive charge and is selected from the group consisting of,

- (i) a substituted or unsubstituted, saturated or unsaturated, linear, branched or cyclic hydrocarbon chain of 1 to 22 carbons;
- (ii) a substituted or unsubstituted, saturated or unsaturated, linear, branched or cyclic hydrocarbon chain of 1 to 22 carbons including at least one heteroatom selected from the group consisting of nitrogen, oxygen, sulfur, phosphorus and halogen;
- 5 (iii) radicals of formula ( $R''-C=O$ ) wherein  $R''$  is a substituted or unsubstituted, saturated or unsaturated, linear, branched or cyclic hydrocarbon chain of 1 to 22 carbons; and
- (iv) radicals of formula ( $R''-C=O$ ) wherein  $R''$  is a substituted or unsubstituted, saturated or unsaturated, linear, branched or cyclic hydrocarbon chain of 1 to 22 carbons including at least one heteroatom selected from the group consisting of
- 10 nitrogen, oxygen, sulfur, phosphorus and halogen.

The positive charge on the  $R^+$  substituent of the phospholipid imparts a zwitterionic character to the compound. This zwitterionic character supplies the surface

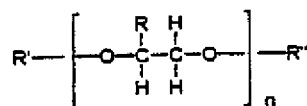
15 active properties that are important to achieve the desired properties of the softening composition of the present invention. Many commercially available phospholipids also contain some quantity of fatty oil and fatty acids. While not essential to the object of this invention, they will serve to impart additional lubricating functionality.

Preferred compounds suitable as a phospholipid for the present invention

20 include phosphatidylcholine (lecithin), hydroxylated phosphatidylcholine, phosphatidylethanolamine, hydroxylated phosphatidylethanolamine,

phosphatidylserine, hydroxylated phosphatidylserine, phosphatidylinositol, hydroxylated phosphatidylinositol, and mixtures thereof.

The non-ionic surfactants suitable for the present invention are based on substituted polyethylene oxide, polypropylene oxide and mixtures thereof. The present invention includes from about 10 to about 85 percent by weight of a non-ionic surfactant. The non-ionic surfactants suitable for the present invention are described by Formula II,



Formula II

wherein n is an integer from 1 to 1000; R is independently selected for each n from the group consisting of:

- (i) hydrogen; and
- (ii) methyl;

R' and R'' are independently selected from the group consisting of:

- (i) hydrogen;
- (ii) a substituted or unsubstituted, saturated or unsaturated, linear, branched or cyclic hydrocarbon chain of 1 to 22 carbons;
- (iii) a substituted or unsubstituted, saturated or unsaturated, linear, branched or cyclic hydrocarbon chain of 1 to 22 carbons comprising at least one heteroatom selected from

the group consisting of nitrogen, oxygen, sulfur and halogen;

(iv) radicals of formula ( $R'''-C=O$ ) wherein  $R'''$  is a substituted or unsubstituted, saturated or unsaturated, linear, branched or cyclic hydrocarbon chain of 1 to 22 carbons;

- 5 (v) radicals of the formula ( $R'''-C=O$ ) wherein  $R'''$  is a substituted or unsubstituted, saturated or unsaturated, linear, branched or cyclic hydrocarbon chain of 1 to 22 carbons comprising at least one heteroatom selected from the group consisting of nitrogen, oxygen, sulfur and halogen.

Examples of  $R'$  and  $R''$  components suitable as described above include, but are  
10 not limited to, ethyl propyl, butyl, oleyl, lauryl, and nonylphenolyl. Examples of commercially available non-ionic surfactants include: Igepal CO-430, and Igepal CO-630 available from GAF Corporation, New York, NY; and Triton X-45, Triton X-114, and Triton X-100 available from Rohm and Haas Company, Philadelphia, Pennsylvania.

A variety of lubricant compositions are suitable for the lubricant portion of the  
15 present invention. The present invention includes up to about 40 percent by weight of the lubricant, preferably about 0.1 to about 6 percent by weight. Preferred lubricants include esters of monoalcohols, dialcohols, polyols, polyethylene glycols, polypropylene glycols, and mixed polyethylene - polypropylene glycol. The glycols and alcohols may be either naturally occurring or of synthetic origin. Preferred polyols  
20 include trimethylolpropane, glycerol and pentaerythritol. Esters are, by definition, chemical compounds that result from the reaction of an organic acid with an organic alcohol. The preferred esters are those derived from acids having side chains selected

from the group consisting of saturated, unsaturated, linear and branched alkyl chains from six to twenty carbons. Long chain monoalcohols having from eight to thirty carbons and fatty alcohols having from ten to thirty carbons are also suitable lubricants for the present invention. The most preferred lubricants are selected from the group consisting of castor oil, olive oil, hexadecanol, methyl tallowate, glycerol, glycerol monostearate, lanolin, decanol and octadecanol.

The softener composition of the present invention is diluted to an appropriate concentration to allow the softener composition to be applied to the cellulose fiber up to 40 pounds of phospholipid per ton of dry pulp. That is, up to 40 pounds of phospholipid per ton of dry pulp could be retained on the final dry paper product.

The following examples will serve to illustrate the unique, and beneficial performance obtained with regard to paper softening, strength, and absorbency, from the combination of phospholipids, non-ionic surfactants and lubricating additives, described according to the present invention.

#### 15. Examples

While not wishing to be bound by a theory of operation, the softness of paper tissue and towel products can be evaluated for their softness properties by use of various factors, all of which are utilized to assess the softness of the product as it may relate and be perceived by the consumer. These measurements include the following:

20. Modulus of Elasticity - defined as the slope of the secant of the graph derived from force vs. stretch % data. Generally considered the lower the force per unit stretch, the greater the flexibility of the sheet.

Shear Strength - Measures the shear stiffness of the test tissue or toweling at a shear angle of 2°, with measurements taken at 0.5 and 5°. A lower shear strength reflects a greater degree of flexibility in the sheet.

5      Bending Strength - The bending test measures the bending strength of the sheet, as well as the moment of hysteresis. A lower bending strength reflects a greater degree of flexibility in the paper sheet.

10      Surface Properties - The surface friction obtained between the paper sheet surface and a u-shaped piano wire, in a sliding contact under constant load and speed is obtained and used to assess the surface friction properties of the sheet and relate this to the hand feel or tactile softness of the sheet. Also under similar conditions of test the surface profile, or roughness of the paper sheet is obtained and is also used as a measure of the surface softness of the paper sheet.

15      Burst Strength - The mullen strength of the sheet. The burst strength is also used to assess the effects of chemical softening compositions on the bulk flexibility of the treated paper.

Kamus Energy - The pulp web is defiberized to asses the energy to remove fibers from the web. The softened pulps would have a lower energy than non-softened pulp.

20      Prior to performance testing, all sheets were conditioned in a climate controlled room at from 45 to 55 percent relative humidity and a temperature from about 71°F to

about 76°F for a minimum of 15 hours. All sheets prepared for each battery of comparative tests were made within the same eight hour period.

The following test procedures were used for the performance tests described below:

5     Absorbance

All absorbance tests were performed in a climate controlled room at from 45 to 55 percent relative humidity and a temperature from 71°F to about 76°F. A sheet strip 1 inch wide and 2.5 inches high was cut from the test sheet and suspended in 100 ml of tap water contained in a 400 ml beaker such that the bottom 0.5 inches of the sheet  
10     was below the water surface. The beaker was left uncovered. The time required for the water to be absorbed from a line 0.5 inches above the water surface to a line 1.5 inches above the water surface was recorded. In a similar manner, oil absorbance was measured by suspending a sheet strip 1 inch wide and 2.5 inches high, cut from the test sheet, in 100 ml of soybean oil contained in a 400 ml beaker such that the bottom  
15     0.5 inches of the sheet was below the oil surface. The time required for the oil to be absorbed from a line 0.25 inches above the water surface to a line 0.5 inches above the water surface was recorded.

Tensile Strength

A sheet strip 1 inch wide and 5 inches high was cut from the test sheet and  
20     placed between the jaws of a tensile testing instrument manufactured by Twining-Albert Instrument Company. The jaws were positioned three inches apart. A 200 pound load cell was used to measure the peak tensile strength of the sheet in pounds per

square inch. The equipment was set with a load range of 10%, a pulling speed of 2 inches per minute, and a high break sensitivity. Wet tensile tests were performed in a similar manner. Immediately prior to beginning the wet tensile test, the sheet strip was saturated with tap water at the midpoint by drawing a wet 1/8 inch wide brush across the sheet, perpendicular to the pulling direction, resulting in a band of saturation across the sheet that was approximately 0.25 inches wide.

#### Example 1

A softener composition was prepared by blending Centrolene A and a hydroxylated soya lecithin available from Central Soya, Inc. with various non-ionic surfactants listed in Table 1 and Table 2, available from Emery Corporation, Cincinnati, OH, and Chemax, Inc at weight ratios required to give non-ideal mixing, and thus synergistic reduction in dry tensile strength that is not seen with lecithin or the non-ionic surfactants individually.

Non-ideal mixing can arise in systems where different surfactants are used, wherein the micelles formed are comprised of mixtures of surfactants, and the resulting electrostatic interactions of the hydrophilic "head" groups can give synergistic effects on the emulsifying properties of the system, often giving substantially lower critical micelle concentrations and interfacial tensions than would be expected based on the properties of unmixed surfactants. Non-ideal mixing is seen by a deviation from the linear relationship of the ratio of the critical micelle concentration to mole fraction obtained with ideal mixing, which is obtained when mixed micelles are not formed.

A batch pulp slurry was prepared with pulp and softener composition concentrations in water that are equivalent to those normally found in the pulp slurry of a conventional tissue or towel paper production machine. A pulp slurry was prepared by mixing 497.45 grams of tap water, 2.45 grams of conventional dry pulp and 0.05 grams of the polyamide-epichlorohydrin resin (Hercules 557H which is a 12% by weight Kymene™ solution wet strength additive available from Hercules, Wilmington, DE for 15 minutes at 60 rpm in a 1000 ml beaker. The slurry was added to a head box measuring 10.5 inches high by 8.0 inches wide by 8.0 inches deep. A screen of approximately 100 mesh was affixed to the bottom of the box to allow for gravity filtration of the liquid through the screen while retaining a wet web of pulp on the top surface of the screen. After the water was drained from the web, a solution of 0.25 percent by weight of the solids components of the softener composition in water was sprayed onto the web using a sprayer available from Badger Air -Brush Co., Franklin Park, IL. Spraying was continued to deliver 0.00625 grams of softener per 2.5 grams of dry sheet. The liquid was allowed to continuously drain through the web while the composition was spray applied. After spraying was completed, the screen, with the web on its upper surface, was removed from the head box. The web and screen were placed, undisturbed, between two sheets of drylap and felt, and pressed through a roller stand manufactured by Adirondack Machine Corp. No additional pressure was applied to the rolls. The sheet formed from the web was then removed from the screen and dried to a constant weight of 2.5 grams using a drum manufactured by Dayton Electronic Mfg. Co. The results of the tests demonstrate that the combination of

hydroxylated soya lecithin and non-ionic surfactant when applied to the web, resulted in the formation of a web with lower dry tensile strength, and higher wet tensile strength, water absorption and oil absorption than the control compositions which included untreated pulp, pulp with lecithin alone, and pulp with polyethylene glycols alone.

Table 1

Test Results: Effects on Wet and Dry Tensile			
Softener Compositions	Dry Tensile (Lb Ft)	Wet Tensile (Lb Ft)	Tensile (Wet:Dry ratio)
15% lecithin + 85% PEG 400 Diester	7.15	1.66	23.2
15% lecithin + 85% PEG 200 Dioleate	7.01	1.78	25.4
30% lecithin + 70% PEG 200 Dilaurate	7.06	1.45	19.8
<b>Controls</b>			
Untreated	10.43	2.1	20.1
Lecithin (100%)	8.68	1.63	18.8
PEG 400 Diester (100%)	7.44	1.33	17.9
PEG 200 Dioleate (100%)	7.76	1.73	22.3
PEG 200 Dilaurate (100%)	7.57	1.67	21.8

Table 2

Test Results: Oil/Water Absorbency		
Softener	Water Absorbency Rate (sec)	Oil Absorbency Rate (sec)
Untreated	56	36.1
100% Hydroxylated Lecithin	43.9	32.5
15% Hydroxylated Lecithin + 85% PEG 400 Diester	41.4	31.2
15% Hydroxylated Lecithin + 85% PEG 200 Dioleate	31.0	25.8
30% Hydroxylated Lecithin + 70% PEG 200 Dilaurate	31.00	31.8

**Example 2**

The following example is provided to demonstrate the effect of the lubricant additive in further enhancing performance of the softening compositions described in the present invention. A batch pulp slurry was prepared with pulp and softener composition concentrations in water that are equivalent to those normally found in the pulp slurry of a conventional tissue or towel paper production machine. Experimental softening compositions were prepared by adding 7% by weight of a lubricant additive listed in Table 3 and Table 4 to a blend of 78% by weight of polyethylene glycol 200 monooleate (available from Chemax, Inc.) and 15% by weight of Centrolene A. A pulp slurry was prepared by mixing 497.42 grams of tap water, 2.48 grams of conventional dry pulp and 0.10 grams of Hercules 557H; (a 12% by weight Kymene™ solution wet strength additive available from Hercules) for 15 minutes at 60 rpm in a 1000 ml

15

### Effects of Lubricant Additives on Dry Strength Reduction

20

Lubricant Additive	Dry Tensile Strength (lb.ft.)	% Decrease over Untreated
Hexadecanol	6.82	26.0
Glycerol Monostearate	6.97	24.4
Methyl Tallowate	6.99	24.2
Decanol	7.01	24.0
Castor Oil	7.07	23.3
Olive oil	7.26	20.3
None	7.36	20.2

Table 4

Effects of Lubricant Additives on Wet Strength Reduction

Softener Composition: 78% by weight Polyethylene glycol monooleate  
 15% by weight Hydroxylated Soya Lecithin  
 7% by weight Lubricant Additive

Lubricant Additive	Wet Tensile Strength (lb.ft.)	Wet/Dry Tensile Ratio
None	1.55	21%
Castor Oil	2.01	28%
Methyl Tallowate	1.54	22%

The following non-limiting examples will serve to describe a suitable method for preparing an effective softener composition according to the present invention

Example 3

Approximately 17.5 percent by weight of polyethylene glycol 200 dilaurate, and 7.5 percent by weight of glycerol (available from Unichema International) were added to 7.5 percent by weight of Centrolene A. The mixture was agitated and heated to a

temperature of 140°F where it was maintained under agitation for 60 minutes. The mixture was added to 67.1 percent by weight of distilled water at 75°F and 0.4 percent by weight of Tektamer 38-LV ( a biocide from Calgon Corporation) and agitated for 30 minutes. At the end of 30 minutes, the mixture was cooled to 75°F over a 120 minute period. The resultant composition was a 32.9% by weight oil in water emulsion of the desired softening composition.

#### Example 4

Approximately 15.0 percent by weight polyethylene glycol 200 monooleate was added to 3.0% by weight of Centrolene A. The mixture was agitated and heated to 140°F where it was maintained under agitation for 30 minutes. Castor oil FCC grade (2.0 percent by weight) was added and mixed for an additional 30 minutes. The mixture was added to 79.6 percent by weight distilled water at 75°F and 0.4 percent by weight of Tektamer 38-LV (a biocide available from Calgon Corporation) and agitated for 120 minutes. At the end of 120 minutes, the mixture was cooled to 75°F with agitation over a 60 minute period. The resultant composition was a 20.4 percent by weight oil in water emulsion of the desired softening composition.

#### Example 5

Approximately 5.25 percent by weight of Lantrol AWS 1692 (a propoxylated and ethoxylated lanolin oil available from Henkel Corporation) was added to 64.60 percent by weight of distilled water at 75°F. The mixture was agitated for 15 minutes. In a separate vessel, 10.5 percent by weight Centrolene A was added to 19.25 percent by weight polyethylene glycol dilaurate (Emerest 2622 available from Henkel Corp). The

Centrolene A and Emerest 2622 mixture was agitated and heated to 140°F where it was maintained for 15 minutes. The Centrolene A and Emerest 2622 were added to the water and lanolin oil mixture. Tektamer 38-LV from Calgon Corporation was added in an amount of 0.4% by weight and the resultant mixture was agitated for 30 minutes. At the end of 30 minutes the mixture was cooled to 75°F over a 120 minute period. The resultant composition was 35.4 percent by weight oil in water emulsion of the desired softening composition.

#### Example 6

A softer towel composition can be produced according to the following steps. A towel product is produced on a paper machine using a Fourdrinier type table and a Yankee Dryer. The sheet is creped off the Yankee Dryer with a doctor blade. A 3 percent by weight pulp slurry (a blend of virgin and recycled fiber) is diluted, at the fan pump, to a head box consistency of 0.2% by weight. This slurry is used to form the sheet on the Fourdrinier wire. The resulting sheet is pressed and transferred to the Yankee Dryer and dried to 6% moisture by weight. A creping adhesive and release agent are applied to the Yankee drier to control the creping.

The softening composition from Example 3 is added to the inlet side of the fan pump at a rate of 10 to 40 lbs of softening composition per ton of air dried pulp. Prior to addition to the pulp slurry, the softening composition is diluted in line with clean water, at a temperature of 70°F to 110 °F. An in line static type mixer is used to assure good mixing. The dilution ratio is 50 parts white water by volume and 1 part softener composition by volume. The towel produced with the softening composition has a softer

hand feel, more bulk and displays better drape properties.

Example 7

A softer fluff pulp composition can be produced according to the following steps.

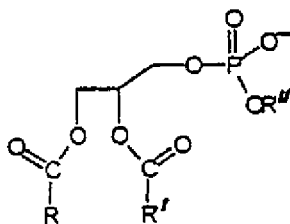
A fluff pulp product for use in diapers is produced on a pulp machine which used a  
5 Fourdrinier type table and Flakt dryer. A 3 percent by weight pulp slurry is diluted at  
the pulp machine fan pump to 1 percent by weight consistency and pumped to the head  
box. The sheet is formed on a Fourdrinier wire, pressed to 40 percent moisture by  
weight, and sent through a Flakt dryer system and dried to 8% moisture by weight.

The softening composition from Example 4 is added to the 3% by weight pulp  
10 slurry prior to dilution with white water at the fan pump. The softening composition is  
added at a rate of 5 to 10 pounds softening composition per ton of air dried pulp. Prior  
to addition, the softening composition is diluted with 100 parts water by volume and 1  
part softener composition by volume with clean water and mixed in a static-type mixer.  
The temperature of the dilution water is maintained at 70°F to 110°F to assure a stable  
15 emulsion. The softener composition is diluted so that it is distributed evenly on the  
pulp fibers. The resulting fluff pulp has a softer hand feel and requires less energy to  
produce. It also does not display losses in its rate of water absorption or capacity of  
water absorption.

The present invention may be embodied in other specific forms without departing  
20 from the spirit or essential attributes thereof and, accordingly, reference should be  
made to the appended claims rather than to the foregoing specification as the indicated  
scope of the invention.

We claim:

1. A method for producing a softened cellulose fiber-based material comprising, treating the cellulose fiber with a composition comprising a phospholipid and a non-ionic surfactant.
2. A method as in claim 1, wherein said composition further comprises a lubricating additive.
3. A method as in claim 1, wherein said composition comprises from 10 to 85 percent by weight of said phospholipid, from 10 to 85 percent by weight of said non-ionic surfactant on a dry basis.
4. A method as in claim 2, wherein said composition comprises from 10 to 85 percent by weight of said phospholipid, from 10 to 85 percent by weight of said non-ionic surfactant, and up to 40 percent by weight of said lubricating additive on a dry basis.
5. A method as in claim 1, wherein said phospholipid is represented by the formula (I)



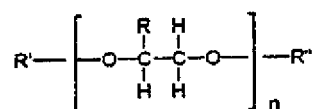
Formula I

wherein R and R' are independently selected from the group consisting of

- (i) a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 6 to 22 carbons;
- (ii) a substituted, unsubstituted, saturated, unsaturated, linear, branched, and cyclic hydrocarbon chain of 6 to 22 carbons comprising at least one heteroatom selected from the group consisting of nitrogen, oxygen, sulfur, phosphorus and halogen; and, R<sup>n</sup> contains at least one positive charge and is selected from the group consisting of,
- (i) a substituted, unsubstituted, saturated, unsaturated, linear, branched, and cyclic hydrocarbon chain of 1 to 22 carbons;
- (ii) a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 1 to 22 carbons comprising at least one heteroatom selected from the group consisting of nitrogen, oxygen, sulfur, phosphorus and halogen;
- (iii) radicals of formula (R<sup>m</sup>-C=O) wherein R<sup>m</sup> is a substituted, unsubstituted, saturated, unsaturated, linear, branched, and cyclic hydrocarbon chain of 1 to 22 carbons; and
- (iv) radicals of formula (R<sup>m</sup>-C=O) wherein R<sup>m</sup> is a substituted, unsubstituted, saturated, unsaturated, linear, branched, and cyclic hydrocarbon chain of 1 to 22 carbons comprising at least one heteroatom selected from the group consisting of nitrogen, oxygen, sulfur, phosphorus, and halogen.
6. A method as in claim 1, wherein said phospholipid is selected from the group consisting of phosphatidylcholine (lecithin), hydroxylated phosphatidylcholine, phosphatidylethanolamine, hydroxylated phosphatidylethanolamine, phosphatidylserine, hydroxylated phosphatidylserine, phosphatidylinositol and hydroxylated phosphatidylinositol.

7. A method as in claim 6, wherein said phospholipid is hydroxylated lecithin.
8. A method as in claim 1, wherein said phospholipids has an HLB value from 6 to 18
9. A method as in claim 1, wherein said non-ionic surfactant is represented by the formula

5



Formula II

- 10 wherein n is an integer from 1 to 1000; R is independently selected for each n from the group consisting of:
- (i) hydrogen; and
  - (ii) methyl; and
- R' and R'' are independently selected from the group consisting of:
- 15 (i) hydrogen;
- (ii) a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 1 to 22 carbons;
  - (iii) a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 1 to 22 carbons comprising at least one heteroatom selected from
- 20 the group consisting of nitrogen, oxygen, sulfur and halogen;
- (iv) radicals of formula (R'''-C=O) wherein R''' is a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 1 to 22 carbons; and

(v) radicals of formula  $(R''-C=O)$  wherein  $R''$  is a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 1 to 22 carbons comprising at least one heteroatom selected from the group consisting of nitrogen, oxygen, sulfur and halogen.

- 5 10. A method as in claim 9, wherein said non-ionic surfactant is selected from the group consisting of polyethylene glycol dioleate, polyethylene glycol dilaurate, polypropylene glycol dioleate, polypropylene glycol dilaurate, polyethylene glycol monooleate, polyethylene glycol monolaurate, polypropylene glycol monooleate and polypropylene glycol monolaurate.
- 10 11. A method as in claim 2, wherein said lubricant is selected from the group consisting of natural esters, synthetic esters, monoalcohols having from eight to thirty carbons and fatty alcohols having from ten to thirty carbons.
12. A method as in claim 11, wherein said lubricant is a polyol ester.
13. A method as in claim 11, wherein said lubricant is an ester of a mono alcohol.
- 15 14. A method as in claim 11, wherein said lubricant is selected from the group consisting of castor oil, olive oil, hexadecanol, methyl tallowate, glycerol, glycerol monostearate, lanolin, decanol and octadecanol.
15. A method as in claim 1, wherein said composition is added to pulp.
16. A method as in claim 15, wherein said composition is added to the head box of a
- 20 paper machine.
17. A method as in claim 15, wherein said composition is added to a machine chest.
18. A method as in claim 15, wherein said composition is added to a stuff box.

19. A method as in claim 1, wherein said composition is added subsequent to the formation of a wet-laid web.
20. A method as in claim 1, wherein said composition is added subsequent to drying of the web.
- 5 21. A method as in claim 1, wherein said composition is added prior to debonding.
22. A method as in claim 1, wherein said composition is added during debonding.
23. A softened cellulose fiber-based product, prepared by the process of claim 1.
24. A softened cellulose fiber-based product, prepared by the process of claim 2.
25. A softened cellulose fiber-based product, prepared by the process of claim 3.
- 10 26. A softened cellulose fiber-based product, prepared by the process of claim 4.
27. A softened cellulose fiber-based product, prepared by the process of claim 5.
28. A softened cellulose fiber-based product, prepared by the process of claim 6.
29. A softened cellulose fiber-based product, prepared by the process of claim 7.
30. A softened cellulose fiber-based product, prepared by the process of claim 8.
- 15 31. A softened cellulose fiber-based product, prepared by the process of claim 9.
32. A softened cellulose fiber-based product, prepared by the process of claim 10.
33. A softened cellulose fiber-based product, prepared by the process of claim 11.
34. A softened cellulose fiber-based product, prepared by the process of claim 12.
35. A softened cellulose fiber-based product, prepared by the process of claim 13.
- 20 36. A softened cellulose fiber-based product, prepared by the process of claim 14.
37. A softened cellulose fiber-based product, prepared by the process of claim 15.
38. A softened cellulose fiber-based product, prepared by the process of claim 16.

39. A softened cellulose fiber-based product, prepared by the process of claim 17.

40. A softened cellulose fiber-based product, prepared by the process of claim 18.

41. A softened cellulose fiber-based product, prepared by the process of claim 19.

42. A softened cellulose fiber-based product, prepared by the process of claim 20.

5 43. A softened cellulose fiber-based product, prepared by the process of claim 21.

44. A softened cellulose fiber-based product, prepared by the process of claim 22.

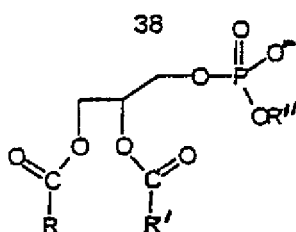
45. A composition for producing a softened cellulose fiber-based material comprising a phospholipid and a non-ionic surfactant.

46. A composition in claim 45, wherein said composition further comprises a lubricating additive.

47. A composition as in claim 46, wherein said composition comprises 10 to 85 percent by weight of said phospholipid and from 10 to 85 percent by weight of said non-ionic surfactant on a dry weight basis.

48. A composition as in claim 46, wherein said composition comprises from 10 to 85 percent by weight of said phospholipid, from 10 to 85 percent by weight of said non-ionic surfactant and up to 40 percent by weight of said lubricating additive on a dry basis.

49. A method as in claim 45, wherein said phospholipid is represented by the formula (i)



Formula I

wherein R and R' are independently selected from the group consisting of

- (i) a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 6 to 22 carbons;
- (ii) a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 6 to 22 carbons comprising at least one heteroatom selected from the group consisting of nitrogen, oxygen, sulfur, phosphorus and halogen; and, R'' contains at least one positive charge and is selected from the group consisting of
  - (i) a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 1 to 22 carbons;
  - (ii) a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 1 to 22 carbons comprising at least one heteroatom selected from the group consisting of nitrogen, oxygen, sulfur, phosphorus and halogen;
  - (iii) radicals of formula (R''-C=O) wherein R'' is a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 1 to 22 carbons; and
  - (iv) radicals of formula (R''-C=O) wherein R'' is a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 1 to 22 carbons comprising at least one heteroatom selected from the group consisting of nitrogen,

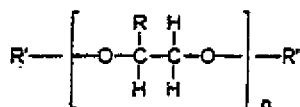
oxygen, sulfur, phosphorus and halogen.

50. A composition as in claim 1, wherein said phospholipid is selected from the group consisting of phosphatidylcholine (lecithin), hydroxylated phosphatidylcholine, phosphatidylethanolamine, hydroxylated phosphatidylethanolamine, phosphatidylserine, hydroxylated phosphatidylserine, phosphatidylinositol and hydroxylated phosphatidylinositol.

51. A composition as in claim 50, wherein said phospholipid is hydroxylated lecithin.

52. A composition as in claim 45, wherein said phospholipid has an HLB value from 6 to 18.

53. A composition as in claim 45, wherein said non-ionic surfactant is represented by the formula (II)



15

Formula II

wherein n is an integer from 1 to 1000; R is independently selected for each n from the group consisting of:

- (i) hydrogen; and
- (ii) methyl; and

- 20 R' and R'' are independently selected from the group consisting of:

- (i) hydrogen;
- (ii) a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic

hydrocarbon chain of 1 to 22 carbons;

(iii) a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 1 to 22 carbons comprising at least one heteroatom selected from the group consisting of nitrogen, oxygen, sulfur and halogen;

5 (iv) radicals of formula  $(R''-C=O)$  wherein  $R''$  is a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 1 to 22 carbons; and

(v) radicals of formula  $(R''-C=O)$  wherein  $R''$  is a substituted, unsubstituted, saturated, unsaturated, linear, branched and cyclic hydrocarbon chain of 1 to 22 carbons comprising at least one heteroatom selected from the group consisting of nitrogen,

10 oxygen, sulfur and halogen.

54. A composition as in claim 53, wherein said non-ionic surfactant is selected from the group consisting of polyethylene glycol dioleate, polyethylene glycol dilaurate, polypropylene glycol dioleate, polypropylene glycol dilaurate, polyethylene glycol monooleate, polyethylene glycol monolaurate, polypropylene glycol monooleate and

15 polypropylene glycol monolaurate.

55. A composition as in claim 46, wherein said lubricant is selected from the group consisting of natural esters, synthetic esters, monoalcohols having from eight to thirty carbons and fatty alcohols having from ten to thirty carbons.

56. A composition as in claim 55, wherein said lubricant is a polyol ester.

20 57. A composition as in claim 55, wherein said lubricant is an ester of a mono alcohol.

58. A composition as in claim 55, wherein said lubricant is selected from the group consisting of castor oil, olive oil, hexadecanol, methyl tallowate, glycerol, glycerol

monostearate, lanolin, decanol and octadecanol.

59. A softened cellulose fiber-based material including a composition comprising a phospholipid and a non-ionic surfactant.

60. A softened cellulose fiber-based material as in claim 59, wherein said material  
5 further comprises a lubricating additive.

61. A softened cellulose fiber-based material as in claim 59, wherein said phospholipid is selected from the group consisting of phosphatidylcholine (lecithin), hydroxylated phosphatidylcholine, phosphatidylethanolamine, hydroxylated phosphatidylethanolamine, phosphatidylserine, hydroxylated phosphatidylserine,  
10 phosphatidylinositol and hydroxylated phosphatidylinositol.

62. A softened cellulose fiber-based material as in claim 59, wherein said non-ionic surfactant is selected from the group consisting of polyethylene glycol dioleate, polyethylene glycol dilaurate, polypropylene glycol dioleate, polypropylene glycol dilaurate, polyethylene glycol monooleate, polyethylene glycol monolaurate,  
15 polypropylene glycol monooleate and polypropylene glycol monolaurate.

63. A softened cellulose fiber-based material as in claim 60, wherein said lubricant is selected from the group consisting of natural esters, synthetic esters, monoalcohols having from eight to thirty carbons and fatty alcohols having from ten to thirty carbons.

64. A method as in claim 1, wherein said phospholipid is added to the cellulose  
20 fibers at a loading of up to about 40 pounds phospholipid per ton of dry pulp.

65. A method as in claim 1, wherein said non-ionic surfactant is added to the cellulose fibers at a loading of up to about 40 pounds non-ionic surfactant per ton of dry pulp.

# INTERNATIONAL SEARCH REPORT

International Application No. <b>PCT/US 97/02423</b>		
<b>A. CLASSIFICATION OF SUBJECT MATTER</b> IPC 6 D21H21/24 D21H17/10 D06M13/292 D06M13/44 D06M13/165 D06M15/53		
According to International Patent Classification (IPC) or to both national classification and IPC		
<b>B. FIELDS SEARCHED</b> Minimum documentation searched (classification system followed by classification symbols) IPC 6 D21H		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practical, search terms used)		
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4 943 350 A (BOGART LARRY ET AL) 24 July 1990 see the whole document ---	1,5,23, 45,59
A	EP 0 365 726 A (SCOTT PAPER CO) 2 May 1990 see the whole document ---	1,5,23, 45,59
A	US 4 766 015 A (NIKOLOFF KOYU ET AL) 23 August 1988 cited in the application see the whole document ---	1,2,20
A	US 2 186 709 A (ROWLAND BEN W) 9 January 1940 ---	
-/--		
<input checked="" type="checkbox"/> Further documents are listed in the continuation of box C. <input checked="" type="checkbox"/> Patent family members are listed in annex.		
* Special categories of cited documents : <div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>*A* document defining the general state of the art which is not considered to be of particular relevance</p> <p>*E* earlier document but published on or after the international filing date</p> <p>*L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>*O* document referring to an oral disclosure, use, exhibition or other means</p> <p>*P* document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>*A* document member of the same patent family</p> </div> </div>		
Date of the actual completion of the international search <b>10 June 1997</b>		Date of mailing of the international search report <b>09.07.97</b>
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax (+31-70) 340-3016		Authorized officer <b>Songy, O</b>

# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/US 97/02423

C/(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4 940 513 A (SPENDEL WOLFGANG U) 10 July 1990 cited in the application -----	

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No  
PCT/US 97/02423

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4943350 A	24-07-90	NONE	
EP 0365726 A	02-05-90	AU 609538 B AU 2502688 A	02-05-91 17-05-90
US 4766015 A	23-08-88	CA 1310006 A	10-11-92
US 2186709 A	09-01-40	NONE	
US 4940513 A	10-07-90	AT 122424 T AU 613765 B AU 3636489 A CA 1328335 A DE 68922529 D DE 68922529 T EP 0347177 A ES 2071658 T JP 2099691 A	15-05-95 08-08-91 21-12-89 12-04-94 14-06-95 21-09-95 20-12-89 01-07-95 11-04-90